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IS : 1060 ( Part III) - 1969
( Reaffirmed 2004 )

# *Indian Standard*

# METHODS OF SAMPLING AND TEST FOR PAPER AND ALLIED PRODUCTS, PART III

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( Including Amendment No. 1 )

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

*July* 1969

# AMENDMENT NO. 1 AUGUST 2004
# TO
# IS 1060 ( PART 3 ) : 1969 METHODS OF SAMPLING AND TEST FOR PAPER AND ALLIED PRODUCTS, PART 3

( *Page* 6, *clause* **7.3.1**, *lines* 3 *and* 6 ) — Substitute '15°' *for* '5°'.

( CHD 15 )

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# METHODS OF SAMPLING AND TEST FOR PAPER AND ALLIED PRODUCTS, PART III

## 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 15 April 1969, after the draft finalized by the Paper Sectional Committee had been approved by the Chemical Division Council.

**0.2** Methods of tests for paper and allied products which are more commonly used have been prescribed in IS : 1060 ( Part I )-1966*. Some of the test methods which are not very commonly used and are for special purposes have been included in IS : 1060 ( Part II )-1960†. Additional test methods which are also for special purposes have been covered in this standard.

**0.3** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960‡.

## 1. SCOPE

**1.1** This standard prescribes methods of test for paper and board for special purposes not covered in IS : 1060 ( Part I )-1966* and IS : 1060 ( Part II )-1960†. The tests prescribed are:

a) Wax absorptiveness,
b) Stiffness,
c) Wax pick number,
d) Surface *p*H,
e) Water penetration,
f) Alkali staining number,
g) Alkalinity, and
h) Copper number.

**1.2** Should any inconsistency exist between the requirements of this standard and those of the standard for an individual material, the latter shall prevail.

## 2. TERMINOLOGY

**2.1** For the purpose of this standard, definitions given in IS : 4661-1968§ shall apply.

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*Methods of sampling and test for paper and allied products, Part I ( *revised* ).

†Methods of sampling and test for paper and allied products, Part II.

‡Rules for rounding off numerical values ( *revised* ).

§Glossary of terms used in paper trade and industry.

## 3. SAMPLING

**3.1** Representative samples for test shall be drawn as prescribed in **3** of IS : 1060 ( Part I )-1966*.

## 4. CONDITIONING

**4.1** Unless otherwise specified all test specimens shall be conditioned in accordance with **5** of IS : 1060 ( Part I )-1966*.

## 5. QUALITY OF REAGENTS

**5.1** Unless otherwise specified, pure chemicals and distilled water ( *see* IS : 1070-1960† ) freshly boiled and cooled, shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## 6. WAX ABSORPTIVENESS

### 6.1 Apparatus

**6.1.1** *Analytical Balance* — sensitive to 0·01 g.

**6.1.2** *Oven* — capable of maintaining a temperature of 103° ± 2°C throughout the interior ( *see* Note under **6.4.4** ).

### 6.2 Materials

**6.2.1** *Blotting Paper* — sheets in the size of 210 × 297 mm, conforming to Grade I of IS : 1396-1960‡.

**6.2.2** *Steel Plates* — sheets of cold rolled steel having smooth, clean and plane surfaces, 150 mm wide, 228 mm long and 6·0 mm thick.

**6.2.3** *Photographic Tray* — 250 × 310 mm.

**6.2.4** *Paraffin Wax* — melting point 54·4° to 55°C.

**6.2.5** *Hot-Plate or Steam-Bath*

**6.3 Test Pieces** — Representative test pieces shall be cut from samples 74 mm wide and 105 mm long. At least four test pieces shall be tested.

### 6.4 Procedure

**6.4.1** Melt the paraffin wax in the tray and adjust the hot-plate temperature so that the molten paraffin is maintained at 70° ± 2°C.

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*Methods of sampling and test for paper and allied products, Part I ( *revised* ).

†Specification for water, distilled quality ( *revised* ).

‡Specification for blotting paper.

**6.4.2** Mark the felt sides of the sheets of the blotting paper. Immerse the sheets of blotting paper in the molten paraffin wax maintained at a temparature of 70° ± 2°C for 30 minutes, then hang them to drain in the oven, maintained at 103°±2°C, for 60 minutes, with the short axis of the sheets parallel to the bottom of the oven. Remove the sheets from the oven and hang them up, separated from each other, to cool at room temperature.

**6.4.3** Trim each of the sheets to 148 × 210 mm cutting in strips along with each edge to ensure even distribution of paraffin in the sheets.

**6.4.4** Weigh each test specimen to the nearest 0·01 g and place it between two sheets of the waxed blotting paper, with the felt side of each sheet of the blotting paper in contact with the test specimen. Place the pad thus formed between two of the steel plates, heated at 103° ± 2°C and withdrawn from the oven immediately prior to use. Place the assembly at once in the oven and heat for 60 minutes at 103° ± 2°C. At the end of this period, remove the assembly from the oven, recondition the test pieces according to **5** of IS : 1060 ( Part I )-1966* and then reweigh them.

NOTE — The chief variances in the use of this method appear to arise from temperature differences in various sections of the oven used. The use of an externally heated, forced-draft oven or of a large electrically heated, thermostatically controlled pulp oven is recommended. To check oven temperatures at various points, the following periodical check is advised:

> A full load of four units of plates is placed in the oven, in the usual test positions, and the maximum number of thermometers are sandwiched between sets of plates for 60 minutes. On the basis of information obtained from these tests, baffle plates may often be inserted to provide uniform temperatures at the various test positions.

**6.5 Calculation**

$$\text{Wax absorptiveness, percent by weight} = \frac{(A - B) \times 100}{B}$$

where

$A$ = weight of the test specimen after wax absorption, and

$B$ = weight of the test specimen before wax absorption.

NOTE — If the cold-rolled, machined steel plates are not available, the use of heavy plates glass is suggested as a substitute. If glass is used, sufficient weight should be added to provide a load of 5·5 kg upon the specimen, to approximate the weight of the steel plate.

## 7. STIFFNESS

### 7.1 Apparatus

**7.1.1** The apparatus shall consist of the following:

a) A sample holder in which the test piece can be clamped;

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*Methods of sampling and test for paper and allied products, Part I ( *revised* ).

b) Means of applying a controlled bending moment to be portion of the test piece protruding from the clamp, the moment arm being 10 mm in length; and

c) Suitable scales to indicate simultaneously the angular deflection of the moment arm and the bending moment applied.

**7.2 Test Pieces** — Representative test pieces 15 mm wide and about 50 mm long shall be cut from the sample in both the machine and the cross directions.

### 7.3 Procedure

**7.3.1** Clamp a test piece in the holder so that the free end of the piece projects 15 to 20 mm. Apply the bending load at such a rate that the moment arm is deflected through 5° in 5 seconds. Note the value of the bending moment and release the load. Repeat the operation three or four times. Record the average bending moment for the test piece in g.cm for a 5° deflection in 5 seconds, neglecting the first reading.

**7.3.2** Cut the next piece in the same direction but deflect it in the opposite sense that is towards or away from the front side as the case may be. Test at least four test pieces in each sense from the machine direction and an equal number from the cross direction.

**7.3.3** Calculate the bending moment in the machine and the cross directions separately and report the average as the stiffness of the sample.

**7.3.4** The value may also be expressed as stiffness factor as follows:

$$\text{Stiffness factor} = \frac{\text{Average stiffness}}{10\,t^3}$$

where

$t$ = thickness in mm.

## 8. WAX PICK NUMBER

### 8.0 General

**8.0.1** *Waxes* — The Dennison standard paper testing waxes are available in a series with graded adhesive powers. The complete series consists of 18 waxes from 2A to 26A, the adhesive strength increasing with the number. Equivalent waxes may be used if available.

### 8.1 Procedure

**8.1.1** Place the test specimen on a smooth surface, such as a hardwood block or a table. The surface should not be a good conductor of heat, and it should not be artificially cooled before the test. The sample sheet should be separated from the block or table by 8 to 10 sheets of paper.

**8.1.2** Select a wax stick and be certain that the end is clean and flat. Heat the end over an alcohol or low gas flame, rotating slowly until several drops of melted wax have fallen. Take care that the wax does not catch fire.

**8.1.3** Quickly place the melted wax end on the surface of the specimen, with firm, but not undue, pressure and withdraw the fingers immediately. Allow 15 minutes for the wax to cool.

**8.1.4** Place a wooden block with a hole over the wax stick, with the stick protruding through the hole in the block. Press the block down firmly with one hand and with the other pull the wax stick from the sheet with a quick jerk at right angles to the paper surface.

**8.1.5** Examine the end surfaces of the wax stick and the specimen. Repeat the test using waxes in advancing numerical order, until the sample surface blisters, breaks, picks or lifts.

**8.1.6** Record as wax pick number, the highest numbered wax which does not disturb the surface of the board.

## 9. SURFACE *p*H

### 9.1 Apparatus

**9.1.0** The following apparatus is required.

**9.1.1** *p*H *Meter* — any standard *p*H meter with glass electrode and a single combination electrode calibrated against standard buffer solutions at two *p*H values ( *see* **9.2.1** ).

### 9.2 Reagents

**9.2.0** The following reagents are required.

**9.2.1** *Buffer Solutions* — two standard solutions, one with *p*H 4 and the other with *p*H 9.

**9.2.2** *Distilled Water* — *p*H 6·0 to 7·2 and carbon dioxide free.

**9.3 Procedure** — Cut a test specimen approximately 50 × 50 mm from the sample sheets drawn as prescribed in **3.1.** Place one large drop of distilled water on the test piece and place electrode in drop, also touching the paper. Take the reading on *p*H meter after about two minutes.

## 10. WATER PENETRATION

**10.1 Apparatus** – The apparatus consists of a metal tube with an internal cross-section of not less than 100 $cm^2$ and not less than 10 cm high, with arrangements to clamp it on the test piece. The apparatus of the type prescribed for cobb test [ *see* **13.2.2** of IS : 1060 ( Part I )-1966* ] is suitable.

**10.2 Procedure** — The paper is subjected to a pressure of 10 cm of water containing one percent Eosin or any other suitable water soluble dye at a temperature of 27° ± 2°C with a filter paper in close contact with the underside of the test piece. At the end of 18 hours, the water is poured out and the filter paper underneath the test piece examined. There should be no staining of the filter paper.

## 11. ALKALI STAINING NUMBER

**11.1 Principle of Method** — The colour of the water extract of the sample, treated with caustic soda solution, is compared with different concentrations of dichromate-congo red solution.

### 11.2 Apparatus

**11.2.1** *Nessler Tubes* — six in number and of 100 ml capacity.

**11.2.2** *Precision Pipette* — 1 ml capacity, graduated to 0·01 ml.

### 11.3 Reagents

**11.3.1** *Potassium Dichromate Solution* — Dissolve 0·25 g of potassium dichromate in a small quantity of distilled water and dilute to 1 litre.

**11.3.2** *Congo Red Solution* — 0·05 percent ( *w/v* ).

**11.3.3** *Sodium Hydroxide Solution* — 1 N.

**11.4 Test Specimen** — The test specimen shall consist of 3·00 g of the paper torn into pieces 0·6 to 1·2 cm square.

### 11.5 Procedure

**11.5.1** Place the test specimen in a 250 ml conical flask, add 50 ml of boiling distilled water and boil for 5 minutes. Decant off the liquid into a 100-ml volumetric flask or graduated cylinder. Add 50 ml of boiling distilled water to the paper in the flask and boil again for 5 minutes. Decant the second liquid into first and make-up to 100 ml. The make-up water used shall also be distilled. Add 25 ml of sodium hydroxide solution to the combined liquids and, after letting it stand for at least 5 minutes, filter, using a fast filter paper, if the insoluble matter is flocculent, or a close filter, if it appears cloudy. Compare the clear filtrate with standards as made in **11.5.2**.

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*Methods of sampling and test for paper and allied products, Part I ( *revised* ).

**11.5.2** To 50 ml of the dichromate solution, add 0·1 ml of the congo red solution and mix well. Place different quantities of this mixed solution, such as 0·2, 0·5, 1·0, 2·0 ml each in separate Nessler tubes, dilute to the 50-ml mark with distilled water and mix.

**11.5.3** Place 50 ml of the filtered alkaline solution ( **11.5.1** ) in another Nessler tube and compare with the standards. Exact matches of tint are not always possible, but distinction in intensity is readily seen. The solutions are best compared by holding the tubes containing them over white paper, but not resting on it, and looking down through the solutions. If none of the standards matches the solution, prepare standards of other strengths and compare them with the solution. As dichromate slowly destroys the congo red colour, the solution should not be mixed until just before use.

**11.6 Report** — Report the number of millilitres of dichromate-congo red solution required to match the tint of the alkaline extract of paper as the alkali-staining number. Express the result to one decimal place.

## 12. ALKALINITY

### 12.1 Reagents

**12.1.1** *Hydrochloric Acid* — 0·02 N.

**12.1.2** *Standard Sodium Hydroxide Solution* — 0·1 N.

**12.2 Procedure** — Place about 5 g but accurately weighed, of paper cut into small pieces in a stoppered bottle containing 250 ml of 0·02 N hydrochloric acid. Allow the mixture to stand for about one hour with occasional shaking. Decant a portion of this solution and titrate a measured quantity against 0·1 N sodium hydroxide solution using methyl orange as indicator. Carry out a blank titration taking the same volume of hydrochloric acid as of the solution taken in previous titration.

### 12.3 Calculation

**12.3.1** Alkalinity ( as $CaCO_3$ ), percent by weight $= \frac{1250\,(A-B)\,N}{V\,W}$

where

$A$ = volume of 0·1 N sodium hydroxide required for blank titration,

$B$ = volume of 0·1 N sodium hydroxide required for extract,

$N$ = normality of sodium hydroxide,

$V$ = volume of the extract taken for titration, and

$W$ = weight in g of sample taken.

## 13. COPPER NUMBER

### 13.0 Principle of the Method

**13.0.1** The copper number of paper is defined as the number of grams of metallic copper in the cuprous oxide resulting from the reduction of copper sulphate, under the conditions of this method, by 100 g of the paper.

**13.0.2** The copper number may be regarded as an index of those impurities in cellulose, such as oxycellulose, hydrocellulose, lignin and sugars, which possess reducing properties. It is valuable for detecting changes accompanying deterioration and may, therefore, be considered as a test for indicating the permanence of paper.

### 13.1 Apparatus

**13.1.1** The apparatus shall consist of the following.

**13.1.1.1** *Grinder* — a grinder that will completely disintegrate the paper without heating or contaminating it. The grinder shall be a Koerner type or its equivalent.

**13.1.1.2** *Bath* — a steam or oil bath maintained at 100° ± 1°C.

### 13.2 Reagents

**13.2.1** *Copper Sulphate Solution* — Dissolve 100 g of copper sulphate crystals ( $CuSO_4,5H_2O$ ) in water and dilute to one litre.

**13.2.2** *Carbonate-Bicarbonate Solution* — Dissolve 350 g of sodium carbonate powder ( $Na_2CO_3,10H_2O$ ) or 129 g of anhydrous sodium carbonate ( $Na_2CO_3$ ) and 50 g of sodium bicarbonate ( $NaHCO_3$ ) in water and dilute to one litre.

**13.2.3** *Molybdophosphoric Acid* — Dissolve 100 g of sodium molybdate crystals ( $NaMoO_4,2H_2O$ ) and 75 ml of phosphoric acid ( 83 percent ) in a mixture of 275 ml of sulphuric acid ( sp gr 1·84 ) and 1·75 litres of water.

**13.2.4** *Sodium Carbonate Solution* — approximately 5 percent ( *w/v* ) in water.

**13.2.5** *Standard Potassium Permanganate Solution ( 0·05 N )* — Dissolve 1·581 5 g of potassium permanganate in water and dilute to one litre in a volumetric flask. Standardize against sodium oxalate as the primary standard.

### 13.3 Procedure

**13.3.1** Completely disintegrate the test pieces in the grinder. Allow the ground material to come to moisture equilibrium with the atmosphere of the balance and weigh a portion of 1·5 g to the nearest 0·10 g.

**13.3.2** Immediately before use, add 5·0 ml of copper sulphate solution to 95 ml of carbonate-bicarbonate solution. Bring the mixture to the boil in 2 min and pour it over 1·5 g of the ground sample in a 125-ml Erlenmeyer flask. Stir well with a glass rod in order to distribute the fibres and to remove air bubbles. Fit the flask with a loosely fitting glass bulb or stopper and submerge completely in a steam-bath at atmospheric pressure. Occasionally, fibres tend to float to the surface, therefore, the flask should be shaken from time to time to redistribute them. Remove the flask from the steam-bath at the end of 3 hours. Filter through an ashless filter paper in a 7·5-cm Buchner funnel using suction. Wash by flooding with 100 ml of sodium carbonate solution at about 20°C and then by flooding with 250 ml of hot water at about 95°C, discarding the filtrates.

**13.3.3** Transfer the fibres and filter paper to a small beaker, add 25 ml of the molybdophosphoric acid solution, and macerate well with a flattened glass rod. Transfer to a Buchner funnel and wash thoroughly with cold water until the blue molybdenum colour is removed from the fibres.

**13.3.4** Dilute the filtrate with water to approximately 700 ml and titrate with 0·05 N potassium permanganate solution to a faint pink colour.

### 13.4 Calculation

**13.4.1** Calculate the copper number as follows:

$$\text{Copper number} = \frac{6{\cdot}36\, V.N}{W}$$

where

$V$ = volume in ml of potassium permanganate solution required for the titration,

$N$ = normality of potassium permanganate solution, and

$W$ = weight in g of the sample taken.

**13.5 Reporting** — The copper number shall be reported on the basis of total fibre content. Not less than two determinations shall be made and the average of the results, rounded off to the nearest 0·1, shall be reported.

**13.6 Reproducibility** — Duplicate determinations should agree within 0·2.

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